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QUARTERLY TECHNICAL PROGRESS REPORT

July - September 1993

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OIL SHALE

TAR SAND

COAL RESEARCH

ADVANCED EXPLORATORY PROCESS TECHNOLOGY

JOINTLY SPONSORED RESEARCH

Under Cooperative Agreement DE-FC21-86MC11076

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1.0 OIL SHALE

1.2 Oil Shale Process Studies

1.2.2 Process Studies (FY 1991 Mod.)

Objectives. The objective of this task is to investigate the use of a shale oil-derived, recycle oil to mediate the transfer of hydrogen to eastern oil shale. This task is composed of three subtasks: (1) the evaluation of a distillate from eastern shale oil as a recycle oil to mediate hydrogen transfer, (2) the determination of the catalytic hydrogenation conditions necessary to regenerate the recycle oil, and (3) the evaluation of the chemical and physical properties of the liquid products from the process to determine their appropriateness as feedstocks for the production of transportation fuels. The objective for this quarter was to complete the topical report.

Accomplishments. A draft of the topical report was prepared and is in review. The report is planned for submittal in October.

2.0 TAR SAND

2.2 Process Development

2.2.1 Recycle Oil Pyrolysis and Extraction (ROPE™) Process (FY 1991)

Objectives. The first objective of this task was to design and initiate fabrication of a modified 6-inch bench-scale unit that included a twin-screw conveyor and a new feed system. This system permits long runs required to evaluate the application of the ROPE™ process to tar sands. The second objective was to develop a process for the treatment of petroleum production wastes commonly termed tank bottom wastes. These wastes are a combination of oil, water, and solids with a water and solids content too high for refinery acceptance of the oil. The objective for the quarter was to complete milestone report 2.2.1C, results of tests on tank bottoms.

Accomplishments. Milestone report 2.2.1C has been completed and submitted. This task is completed.

3.0 COAL RESEARCH

3.2 Coal Combustion

3.2.3 Gasification and Cogeneration (FY 1992)

Objectives. The objective of this research is to select and develop a combustor design and hot-gas cleanup system suitable for use with low-sulfur coal. The objective for this quarter was to complete the milestone report.

Accomplishments. The milestone report is being prepared. The report is planned for submittal in October.

3.3 Integrated Coal Processing Concepts

3.3.4 Coal Coprocessing (FY 1992)

Objectives. The objectives of this research are to define more closely coprocessing conditions that improve the liquid yield through more efficient dispersion of iron-based catalysts and to characterize the acid sites on supported catalysts and their impact on the formation of coke. The objective for the quarter was to complete the topical report.

Accomplishments. A draft of the milestone report was prepared and reviewed in-house. Final revisions were being made to the report. The report is planned for submittal in October.

3.3.6 High-Heating-Rate Process Studies (FY 1992)

Objectives. The objective of this research task is to determine oil yields from the rapid pyrolysis of coal in an entrained flow reactor. The objective for the quarter was to prepare the milestone report.

Accomplishments. The milestone report is being prepared.

3.4 Solid Waste Management

3.4.1 Use of Solid Waste for Chemical Stabilization (FY 1991 and FY 1992)

Objectives. The objective of this research is to determine a use for fly ash, such as that produced as waste during operations at the Dave Johnston Steam Electric Project, that would be economically and environmentally reasonable. Dave Johnston fly ash has been shown to have some affinity for selected organic compounds that have created problems in the environment. The objective for the quarter was to complete the topical report, 3.4.1B.

Accomplishments. The topical report, 3.4.1B has been completed and submitted. This task is completed.

4.0 ADVANCED EXPLORATORY PROCESS TECHNOLOGY

4.1 Advanced Process Concepts

4.1.7 In Situ Model of Pyrolysis (FY 1990)

Objectives. The objectives of this research effort are to predict solute transport and develop new control concepts that incorporate some of the more recent geochemical data obtained from research projects. The objective for the quarter was to complete the topical report.

Accomplishments. The topical report was completed as part of milestone report 4.3.3A. This task is completed.

4.1.9 In Situ Process Modeling (FY 1991)

Objectives. The objectives of this research task are to conduct laboratory simulations and develop a numerical model for the simulation of the steamflood process in fractured reservoirs. The objective for this quarter was to complete the milestone reports.

Accomplishments. Drafts of the reports are being prepared. The reports are planned for submittal in October.

4.2 Advanced Mitigation Concepts

4.2.3 CROWTM Development (FY 1992)

Objectives. The objective of this research is to obtain baseline data that show the effectiveness and environmentally safe use of chemicals to enhance the CROWTM process. The objective for the quarter was to prepare the milestone report.

Accomplishments. A draft of the milestone report is being prepared.

4.2.4 Environmental Treatment of Process Gases (FY 1992)

Objectives. The objective of this research is to optimize vortex combustor design to obtain maximum thermal destruction efficiency at selected temperatures and retention times. The objective for the quarter was to complete the milestone report on the research.

Accomplishments. The milestone report has been completed and submitted. This task is completed.

4.3 Oil and Gas Technology

4.3.1 Enhanced Oil Recovery (FY 1992)

Objectives. The original objective of this research task was to determine the enhancement of oil recovery using steamflooding in conjunction with chemicals or gases and horizontal wells. The approved experimental plan for this task has eliminated the use of horizontal wells. The objectives for this quarter were to complete the experiments and prepare the milestone report.

Accomplishments. Tests have been completed and the report is being prepared. The report is planned for submittal in October.

4.3.3 Thermal Reservoir Simulation (FY 1992)

Objectives. The objective of this research effort is to improve the capabilities of Western Research Institute's (WRI) thermal reservoir simulation model. The objective for the quarter was to prepare the topical report.

Accomplishments. The topical report was completed and submitted. This task is completed.

5.0 JOINTLY SPONSORED RESEARCH

5.1 Occidental Oil Shale, Inc. Demonstration Program Support

This task was completed in previous quarters.

5.2 Investigation of ROPE[®] Process Performed on Sunnyside Tar Sand

This task was completed in previous quarters.

5.3 Organic and Inorganic Hazardous Waste Stabilization

Objective. The objective for this quarter was to prepare the topical report.

Accomplishments. A draft of the topical report was completed and is in review. The report is planned for submittal in October.

5.4 Optimization of Product Yields for the CHARFUEL Process

This task was completed in previous quarters.

5.5 Cold Flow Injector Mixer Project for the CHARFUEL Project

This task was completed in previous quarters.

5.6 CROWTM Field Demonstration with Bell Lumber and Pole

Objectives. The objective of this task is to design, construct, and operate a field demonstration of the CROWTM process technology to treat a site contaminated with organic wastes from the wood treatment process. The objectives for the quarter were to obtain approval of the work plan for the full-scale demonstration test and begin preparing equipment specifications for the test.

Accomplishments. Based on meetings and discussions with the Minnesota Pollution Control Administration (MPCA), modifications to the detailed work plan for the full-scale demonstration have been made and resubmitted. The modified work plan has been tentatively accepted by the MPCA. The permitting approvals have delayed the project significantly. A letter proposal was submitted requesting carryover of the project into the new JSR Cooperative Agreement as a new task. This request was approved.

Procedures. The activities being carried out to accomplish this research task include: review and analyze available site data, develop design parameters from site data and one-dimensional physical simulations of the process using site materials, develop process flow diagrams of surface and subsurface systems, assist in the development and submittal of all project permits required by local, state, and federal agencies, conduct a two-well test to provide additional information on organic displacement and hydraulic confinement, develop detailed design for all injection, production, and water treatment systems, assist in the purchase or lease of all required equipment, monitor the facilities construction, assist in the monitoring of the displacement operations, and evaluate and report on the performance of the displacement process.

Results. The two-well pilot test was successful. Work began on specification of equipment for the full-scale demonstration.

5.7 Development and Validation of a Standard Test Method for Sequential Batch Extraction Fluid

This task was completed in previous quarters.

5.8 PGI Demonstration Project

Status. With no additional funding from the cosponsor, work was redirected to other JSR tasks.

5.9 Mild Gasification of Usibelli Coal

This task was completed in previous quarters.

5.10 Real-Time In-Situ Remote-Sensor Development

This task was completed in previous quarters.

5.11 Enhanced Gravity Drainage in the North Tisdale Reservoir Using Horizontal Wells

This task was completed in previous quarters.

5.12 Solid State NMR Analysis of Powder River Basin Shales

This task was completed in previous quarters.

5.13 Operation and Evaluation of the CO₂ HUFF-N-PUFF Process

Objectives. The objectives of this task are to (1) conduct in situ residual oil saturation determinations, (2) assist in the design, operation, and monitoring of well tests, (3) determine the characteristics of collected fluid samples, (4) assist in the development of phase equilibrium relationships with chemical and thermodynamic properties of selected crude oils, (5) assist in the development of a predictive numerical process model, and (6) assist in the analysis, reporting, and dissemination of collected data. The objective for this quarter was to continue preparation of the final report.

Accomplishments. All testing has been completed and all field samples have been collected and analyzed. The basic numerical model is operational and special functions that incorporate hysteresis, viscous fingering, and wettability and surface tension changes were used in evaluations. Analysis of samples collected for development of phase equilibrium relationships have been completed. The final report is being prepared.

Procedures. Over 1,867 gas and 1,418 fluid samples have been taken from the field tests of the CO₂ Huff-n-Puff process. Routine gas analysis has been performed on approximately 1,675 of the gas samples. Oil-water separation has been completed on approximately 1,418 of the fluid samples with further analysis on approximately 275 of these samples.

Modifications to incorporate hysteresis, viscous fingering, and wettability and surface tension changes into the numerical model have been made and the model is operational.

Results. Preliminary well responses show mixed results as to the success of the CO₂ stimulations. The single well tracer tests have been shown to be invaluable in determining the potential of candidate wells for the CO₂ cyclic stimulation.

5.14 Fly Ash Binder for Unsurfaced Road Aggregates

Objectives. The objectives are to develop and demonstrate the use of Wyoming fly ash in two construction techniques: (1) fly ash stabilization of soils as applied to unpaved roads and (2) fly ash replacement of portland cement in conventional cement treated bases (CTB). The use of fly ash in these construction technologies could potentially result in lower cost construction techniques and provide the ash generators new or expanded markets for Wyoming coal fly ash. The development of commercial enterprises, based on these technologies for the enhanced use of coal power plant fly ash in construction applications, therefore represents both a business and an environmentally attractive option for the state of Wyoming. The objective for the quarter was to continue preparation of the final project reports to the various sponsors.

Accomplishments. Preparation of the final reports is under way.

Procedures. Three reports will cover the testing and demonstration activities.

(1) Laboratory testing of soils, fly ash, and aggregate samples that was concluded under subcontract to the University of Wyoming, Department of Civil Engineering. Fly ash stabilization of unpaved roads laboratory testing examined three additional soils ranging from AASHTO soil types A-1b to A-6 (RS-1, RS-2, RS-5, and RS-7) with two additional fly ashes (Naughton and Laramie River). The results of the durability tests provided an evaluation of whether or not sufficient strength and durability can be realized at these low dosages under the climatic conditions of the Rocky Mountain region. A report covering the CTB testing has been completed by the subcontractor and has been submitted to WRI for review and incorporation into a WRI final report.

(2) Laboratory testing for CTB applications using a scoria aggregate from Reno Junction, Type II portland cement from Mountain Cement and the above mentioned three fly ashes (Dave Johnston, Naughton, and Laramie River). The fly ash replacement of the portland cement ranged from 20% to 75%. Approximately 8% portland cement or cement plus fly ash is used in the CTB mixes. A report covering the CTB testing has been completed by the subcontractor and has been submitted to WRI for review and incorporation into the WRI final report.

(3) Field demonstration of the fly ash treatment of unpaved roads. An interim report covering the fly ash treatment of unpaved roads and, in particular, the demonstration test section in Converse County had been submitted to the U.S. Department of Energy (DOE), PacifiCorp, and STEA for review and approval. It is now being converted into a final technical report on the Glenrock Demonstration.

5.15 Evaluation of Products Recovered from Scrap Tires for Use as Asphalt Modifiers

This task was completed in previous quarters.

5.16 Solid State NMR Analysis of Mesaverde Group, Greater Green River Basin, Tight Gas Sands

Objectives. The objectives of this study are to apply solid-state ^{13}C nuclear magnetic resonance (NMR) to measure changes in the organic structure of petroleum source rocks (kerogens) brought about by laboratory hydrous pyrolysis experiments and by maturation in the natural geologic environment as a result of depth of burial. These data, in conjunction with other analyses and kinetic measurements, will be used by the University of Wyoming, Department of Geology and Geophysics to develop an innovative exploration and production strategy that will optimize the efficient exploitation of tight gas resources in the Mesa Verde Group, Greater Green River Basin, Wyoming. The objective for the quarter was to complete the topical report on the task.

Accomplishments. The topical report has been submitted and approved. This task is completed.

5.17 Flow-Loop Testing of Double-Wall Pipe for Thermal Applications

Objectives. The objectives of this research effort are to develop a numerical model that will predict down-hole steam quality, steam pressure, and temperature, to evaluate InterMountain Pipe Company's double-wall pipe for thermal application, and provide future industrial clients with a fully instrumented flow loop. The objective for the quarter was to complete the final report.

Accomplishments. The final report was completed and submitted in the previous quarter. Revisions were made to address review comments. This task is completed.

5.18 Characterization of Petroleum Residua

Objectives. The objectives of this effort are to develop methods and apply them for characterization of petroleum residua from industry participants. The objectives for the quarter were to complete all remaining work and prepare the final report.

Accomplishments. The analytical size exclusion chromatography and NMR aromaticity work were completed for the fractions and asphaltenes from Residua D, E, and F. The final report was prepared, submitted, and approved. This task is completed.

5.19 Shallow Oil Production Using Horizontal Wells with Enhanced Oil Recovery Techniques

Objectives. The objectives of this task are to demonstrate that enhanced oil recovery techniques can be successfully used with horizontal wells in shallow reservoirs to increase oil production significantly, to validate a numerical model with the use of physical simulations using an implemented enhanced oil recovery process with horizontal wells, and to provide the technical expertise and supervision for the implementation of a pilot test that will use the information generated in the study. The objective for the quarter was to prepare the topical report.

Accomplishments. Preparation of the topical report continued.

Procedures. Production data, gas samples, and temperature data were collected during the test. Injection of air into the formation has been terminated. All wells have been shut-in. Abandonment of the wells will not proceed until the final report is prepared.

Results. Temperature increases in the horizontal production wells were not observed during the air injection phase. Injection of air has been terminated due to the economics of the project. A total of 40 barrels of reservoir crude oil was produced.

5.20 "B" Series Pilot-Plant Tests

This task was completed in previous quarters.

5.21 Surface Process Study for Oil Recovery Using a Thermal Extraction Process

Objectives. The objective of this task is to develop an economic process for shallow oil resources which are not being utilized. The objectives for the quarter were to continue the scale-up design and prepare a preliminary report on the reservoir characterization, physical simulations, and scale-up to field scale.

Accomplishments. The reservoir has been characterized, and the physical simulations of the thermal process and the gravity drainage process have been completed. The process and product evaluations have been completed and the scale-up of the process to field scale is nearly completed. The preliminary report, covering the first five phases of the project, was prepared and sent to the cosponsor and to DOE. The field demonstration unit was fabricated, moved to the field, and tested. The topical report for the field demonstration has been initiated.

Procedures. The phases of the project are: (1) characterization of the preselected target reservoir, (2) physical simulation of the thermal process using reservoir material, (3) physical simulations of the gravity drainage process, (4) process and product evaluation, (5) scale-up of the process to a field-pilot, (6) field demonstration of the process, and (7) project reporting. Thermal processing of reservoir material will be evaluated using existing equipment at WRI. Based on the laboratory simulations, a field-scale demonstration unit will be designed, constructed, and operated at a field location.

5.22 NMR Analysis of Samples from the Ocean Drilling Program

This task was completed in previous quarters.

5.23 Menu Driven Access to the WDEQ Hydrologic Data Management System

This task was completed in previous quarters.

5.24 Oil Field Waste Cleanup Using Tank Bottom Recovery Process

Objectives. The objective of this task is to remediate an oil field waste problem and thereby develop an energy resource that is currently being wasted, discarded, or destroyed. The objective for the quarter was to complete the equipment procurement.

Accomplishments. Preliminary evaluation of the process was accomplished under another program. Based on results of the previous project, a preliminary process schematic and material balance was developed. Equipment procurement was completed.

Procedures. Based on previous laboratory simulation results, a field-scale unit is being designed and constructed. It will be tested and operated in the field by the cosponsor. Reporting will follow testing.

5.25 Remote Chemical Sensor Development

Objectives. The objectives of this research effort are to design, test, and construct prototype field instrumentation for in situ qualitative identification and quantitative determination of selected groundwater pollutants. The objectives for this quarter were (1) to continue the laboratory experiments to develop a new, simple field method to determine diesel fuel and other hydrocarbon materials in soils, (2) to begin evaluation of an ISI-constructed prototype probe for detecting volatile organic compounds in groundwater, and (3) to begin evaluating several commercial down-hole probes for environmental analysis.

Accomplishments. Experimental work on the extraction of diesel fuel from contaminated soils using non fluorocarbon solvents continued. Several solvent systems were tested. One in particular shows promise in selectively extracting the diesel with minimal background. A draft method was written. A laboratory photometer was constructed and tested successfully at ISI. A prototype field unit is being built.

A laboratory electro-optical device was constructed at ISI to measure volatile organic compounds in groundwater. Some experiments were performed to establish the time required to reach vapor equilibrium in headspace, and to establish the response characteristics of the device relative to concentrations of toluene measured by gas chromatography.

Several commercial down-hole probes are being evaluated. Results are being compared with laboratory methods.

The Cooperative Agreement funding this 30-month effort is due to expire on September 30, 1993, 11 months into the project. A proposal to complete the project as a new task under the new JSR Cooperative Agreement was approved.

Procedures. A field analysis product, Lab-in-a-Bag, has been evaluated for its response to diesel fuel and weathered gasoline in sand, silt, and clay soil matrices.

Results. In the diesel fuel extraction work, one solvent system showed promise in selectively extracting the hydrocarbons. Evaluation of the Lab-in-a-Bag showed that this is a good tool for measuring gasoline, but has limited application for less volatile diesel fuel. The evaluation also found that a higher and more uniform signal is obtained by analyzing soils with no water added.

5.26 In Situ Treatment of Manufactured Gas Plant Contaminated Soils Demonstration Program

Objectives. The objectives of this task are to demonstrate and evaluate the CROWTM process and bioremediation to remediate a site contaminated with a dense organic fluid. Hopefully, treatment levels in the field will achieve results that are comparable to prior laboratory findings. The objective for this quarter was to satisfy permitting requirements in order that the construction and operation can proceed.

Accomplishments. The work plan for the field demonstration was drafted and submitted to the U.S. Environmental Protection Agency (EPA) Region 3 office for approval earlier. All environmental and construction permits have been identified and the permitting phase is underway.

A 30% design of the field installation and operating scheme was prepared for submittal to the EPA Region 3 office as part of the EPA site requirements. The Pennsylvania Department of Environmental Resources is also being included in the design, review, and operational requirements of the project. Review comments on the 30% design have been received.

The 95% design, part of the EPA site requirements, for the field demonstration has been essentially completed for submittal to the EPA in mid-October. Procurement of equipment for site operations has been initiated.

Progress on the project has been slowed because of problems encountered in the recharacterization of the site as requested by the EPA. The recharacterization was completed in late June and the work on the field demonstration has resumed. The permitting approvals have delayed the project significantly. A letter proposal was submitted and approved to carryover the project into the new JSR Cooperative Agreement as a new task.

Procedures. The overall task procedures are: (1) develop and submit a detailed work plan, (2) identify all required construction and environmental permits, (3) apply for all permits, (4) prepare and submit for approval a 30% complete design for the field layout and operation, (5) prepare and submit a prefinal design for the field layout and operation (all equipment, well construction, and operating routines will be fully specified), (6) prepare a final project design based on the review comments from the prefinal design submission, (7) procure all required equipment and construct the field facilities, (8) operate the field demonstration test, (9) dismantle the field facility, and (10) analyze the data from the field demonstration and report the results to DOE, EPA, and the cosponsors.

The laboratory results that are being used to design this project are from a completed project sponsored by the EPA's Emerging Technologies Program.

5.27 Solid State NMR Analysis of Mowry Formation Shale From Different Sedimentary Basins

Objectives. The objective of this task is to apply the solid-state ^{13}C NMR techniques of cross polarization with magic-angle spinning (CP/MAS) to measure changes in organic carbon structure of kerogens brought about by laboratory hydrous pyrolysis experiments and by maturation in the natural geologic environment as a result of depth of burial. These data, in conjunction with other analyses, and kinetic measurements will be used to construct a model for reconstructing the diagenetic and maturational history of pressure chambers. Ultimately, this information will become a framework for seismic detection of these pressure chambers. The objectives for the quarter were to acquire samples and perform NMR measurements.

Accomplishments. Samples of Mowry shale from the Big Horn Basin, Wyoming have been acquired. NMR measurements are in progress.

Procedures. Solid-state NMR measurements are made on a Chemagnetics CMX 100/200 solids NMR spectrometer. ^{13}C NMR spectra are obtained using CP/MAS. The ^{13}C measurements are made at a frequency of 25 MHz. This task continues as a new task under the new JSR Cooperative Agreement.

5.28 Solid State NMR Analysis of Naturally and Artificially Matured Kerogens

Objectives. The objective of this research effort is to apply solid-state ^{13}C NMR techniques of cross polarization with magic-angle spinning (CP/MAS) to measure changes in the organic carbon structure of the kerogen in petroleum source rocks brought about by maturation in the natural environment and by artificial maturation in laboratory environments. These data, in conjunction with other geochemical analyses and kinetic measurements, will be used to establish the basic information required for calculating kinetic parameters used in the modeling of the generation of critical compounds in diagenetic systems. The objective for the quarter was to evaluate NMR measurements on source rock samples.

Accomplishments. Data from the hydrous pyrolysis are being analyzed. Results of NMR analyses of hydrous pyrolysis residues and naturally matured coals and shales were presented at the 7th International Conference on Coal Science, Banff, Alberta, September 12-17, 1993.

Procedures. Solid-state NMR measurements are made on a Chemagnetics CMX 100/200 solids NMR spectrometer. ^{13}C NMR spectra are obtained using CP/MAS. The ^{13}C measurements are made at a frequency of 25 MHz. The NMR data are being fit using computer software to provide further discrimination of carbon functional groups. In particular, the aliphatic carbon functionality is being deconvoluted into methylene carbons and carbons in methyl groups attached to aromatic rings and terminating aliphatic chains. Results of this analysis will be useful for modeling the kinetics of oil generation during hydrous pyrolysis.

NMR measurements were made on residue samples from hydrous pyrolysis experiments conducted at temperatures from 290 to 360°C (554 to 680°F) for 72 hr. The aliphatic resonance band was deconvoluted into resonances due to methylene carbons (30 ppm), methyl groups attached to aromatic rings (~20 ppm) and terminal methyl groups on side chains (~16 ppm) using curve-fitting procedures. This provides a further discrimination of carbon functionalities that are prone to produce oil and gas.

Results. During oil generation, the major changes in the aliphatic components are the disappearance of the methylene carbons (30 ppm) and the relative increase in the number of methyl groups attached to aromatic rings. This task continues as a new task under the new JSR Cooperative Agreement.

5.29 Development of an Effective Method for the Clean-Up of Natural Gas

Objectives. The objective of this task is to evaluate the feasibility of using a molecular sieve carbon that is manufactured by the Takeda Chemical Company of Japan as the solid adsorbent in a pressure swing adsorption (PSA) cycle to separate nitrogen from natural gas. Development of a less complex and more cost-effective method for upgrading low-quality natural gas by the use of physical separation processes will require an adsorbent that can function in a simple pressure swing cycle, instead of the pressure/vacuum swing cycle used in currently available physical separation processes. The objective for this quarter was to prepare the topical report.

Accomplishments. In previous quarters, the effects of pressurization rate, depressurization rate, shut-in time between pressurization and depressurization, purge rate and volume, purge and blow-down direction (cocurrent or countercurrent) have been investigated. The use of a 100% methane purge has also been investigated. Preparation of the topical report continued.

Procedures. A small laboratory adsorption column was loaded with molecular sieve carbon and degassed under vacuum. The sieve is a 5 angstrom carbon molecular sieve manufactured by the Takeda Chemical Company of Japan. For a typical test, the column is pressurized at a predetermined rate with a mixed gas consisting of 30% nitrogen and 70% methane, allowed to depressurize, and purged with the feed gas mixture. The effluent from the column is collected and its volume is measured by water displacement. The effluent is collected in increments corresponding to various column pressure ranges and the increments are analyzed by gas chromatography. Product purity, product recovery, and sorbent productivity are calculated from the collected data.

Results. Little effect on separation performance resulted from changing pressurization and depressurization rates or the length of shut-in between pressure changes. Purge rate is not as significant as purge volume. Countercurrent blow-down and purge both result in minor improvement for this separation. The 100% methane purge resulted in a lower recovery of methane from the feed, with no improvement in product purity. The best conditions observed so far result in a product purity of 87%, 61.5% methane recovery, and a sorbent productivity of 12.2 CC/gram/cycle.

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